**Results - abstract**

2021 - Design, construction and testing of new equipment allowing simultaneous use of MW and US - Studies on the influence of US on MW heating

Design, construction and testing of the new MW + US equipment (based on the Labotron system) A complex equipment was designed and manufactured that allows the combined use of ultrasound (US) and microwaves (MW). The microwave component was the Labotron metal reactor (1.5L), equipped with INTLI antenna and 1kW microwave generator. To this metal reactor was coupled an ultrasonic equipment of "cuphorn" type with frequency of 20kHz and power of 200 W (bought by project). To homogenize the reaction mixture, a recirculation pump was also used, whose flow can be controlled. For this equipment were made technical documentation and instructions for use. The testing of this equipment was carried out by determining the microwave and ultrasonic power absorbed by the reactor liquid. The INTLI antenna imposed the correlation of the absorbed power with the reactor liquid level, thus determining the optimal level in the reactor for a safe operation of the equipment. Also for this purpose was made an INTLI stainless steel antenna, as well as a glass sheath for it. This equipment was also introduced in Comsol, including the three-piston adapter "3-stub tuner".

After the implementation of the Labotron equipment in Comsol, the aim was to compare the results obtained experimentally (absorbed powers, calorimetrically determined) with those from the simulations in Comsol. With the introduction of the "3-stub-tuner" adapter in the model, the results obtained in the model were consistent with those obtained experimentally. Studies on the influence of the US on MW heating (both equipment will be used) Two distinct pieces of equipment were used: equipment based on the Labotron system (with a 1.5L metal reactor) and another equipment based on the Miniflow system with a 100 mL glass reactor. On the latter equipment, the US influence on MW heating can be better determined because the system can behave adiabat at small temperature differences. In order to highlight the changes in absorption of microwave energy when the reaction mixture in the reactor is subjected to ultrasound, a measuring device of the network vector analyzer (NPV) type was used. With its help, the reflected power (S11), impedance and VSWR ratio were measured. When determinations were performed under thermal equilibrium conditions between the US bath and the single-mode cavity reactor, reproducible determinations were possible that revealed the effect of US on microwave energy absorption. The determinations were performed on a special type of glass reactor in which the liquid in the US bath is put in direct contact with that in the reactor. The influence of US at different frequencies (580, 864 and 1146 kHz) and different amplitudes on the absorption of microwave energy has been highlighted. It was aimed to identify this effect especially in the absence of cavitation, when the appearance of gas bubbles causes changes in the absorption of microwave energy. This configuration was also introduced in Comsol, highlighting the US and MW fields respectively in the different areas of the equipment.

2022 - Intensification of calcium alginate extraction and synthesis processes in MW and US combined reactor

Intensification of extraction processes of fat-soluble active principles The aim of this study was to use an innovative plant that allows simultaneous application of microwaves and ultrasound to improve the extraction yield of β-carotene from sea buckthorn fruits. The resulting extract is intended to be used as a food supplement, so ethyl esters of fatty acids obtained by transesterification of hemp oil into enzymatic catalysis were used as solvent for extraction, while using ethyl alcohol for pharmaceutical use. The tests performed on the innovative device led to the conclusion that the highest ultrasonic power absorbed by the solvent is achieved when an amplitude of 50% is applied and the reactor is immersed in the ultrasonic bath 2 cm. As regards the study of β-carotene extraction from sea buckthorn fruits, it has been shown that with the conventional extraction method the influence of temperature for the range 50-70 °C is insignificant. After studying the type of heating on the extraction efficiency, it was concluded that all unconventional methods lead to better results compared to the classical method. In the case of ultrasound-assisted extraction alone, the influence of amplitude on the β-carotene content was also verified, noting that higher amounts of bioactive compound are obtained for an amplitude of 50%, its increase to 70% leading to degradation of the compound of interest. Taking into account the type of heating, the most effective method was simultaneous extraction with microwave and ultrasound. For this method, the influence of ultrasound amplitude, plant/solvent ratio and extraction time were also checked. Thus, the best results were obtained for simultaneous microwave and ultrasound assisted extraction at an amplitude of 50%, a microwave power of 15 W, a temperature of 52 °C, a plant/solvent ratio of 1/40, an extraction time of 90 min (mixing of the extraction medium was done mechanically, and the reactor was immersed in the ultrasonic bath 2 cm).

Under the aforementioned conditions, almost 80% of the total amount of β-carotene found in sea buckthorn fruits has been reached. At the same time, it was verified what type of effect leads to the simultaneous use of microwaves and ultrasounds, finding that the effect is not synergistic. Because it is desired to use βcarotene-rich extract as a dietary supplement, its stability over time was also determined, finding that adding a powerful antioxidant (vitamin E) leads to a very good stability over time of β-carotene even after 3 months. Synthesis, characterization and testing of calcium alginate obtained by intensifying the process using MW and/or US The main objective of the research proposed at this stage was the use of ultrasound (US) and microwaves (MW), either separately or together in the preparation of calcium alginate (CaAlg) from sodium alginate to be used in removing Pb(II) and Cd(II) from single-component aqueous solutions and binary solutions. US of different amplitudes (50% and 100%) have been applied to study the effect of power ultrasound on size, surface morphology and heavy metal ion wastewater remedial capacity. The best results were obtained in the case of CaAlg prepared by the US-assisted method of 100% amplitude. When a combined US and MW system was used in the preparation of CaAlg, the results were spectacular: over 50% higher removal efficiencies of heavy metal ions than conventional material were obtained. These green synthesis methods were compared for the first time to determine their influence on CaAlg properties. Exposure to US increased surface roughness/porosity based on acoustic cavitation, and MW produced cracks and voids on the CaAlg surface. This illustrates different values of specific surface area and removal capacity. The combined use of MW with US is efficient to improve the removal capacity of Cd(II) and Pb(II). The Langmuir isotherm model suggested a single-layer adsorption of Cd(II) and Pb(II) on the homogeneous surface of CaAlg. The kinetics of the adsorption process are second-order, indicating a chemical adsorption. Competitive adsorption of Cd(II) and Pb(II) in binary solutions shows that both heavy metal ions prevent removal of the other metal ion. Green synthesis methods can be successfully applied to prepare adsorbents with improved removal capacity that can be used for wastewater treatment.

2023 - Intensificarea proceselor de tratare a biomasei pentru obtinerea de polioli, cu ajutorul MW si US in reactorul combinat

The aim of the research carried out at this stage was to obtain biopolyols from lignocellulosic biomass, which can be used in the manufacture of a multitude of products of great interest to the chemical industry and the general public: polyurethane foams and adhesives

Lignocellulosic biomass, i.e. woodworking waste (sawdust) or agricultural residues (corn cobs) was used to obtain biopolyols through a glycolysis process in acid catalysis or base catalysis. Lignocellulosic wastes reacted using conventional and microwave heating in the presence of acid catalysts type H2SO4 96%, H3PO4 85% and zeolite H-ZSM-5, respectively basic catalysts type NaOH, with a glycolysis agent - diethylene glycol (DEG).

The experimental program involved monitoring reaction parameters such as: biomass:solvent ratio, reaction temperature, reaction time, catalyst type. The influence of these parameters on biomass conversion and biopolyol characteristics (factorial program) was evaluated.

Also, experiments were conducted involving the use of acid or basic catalysts and mixtures of zeolites with homogeneous acid catalysts. The amount of catalyst/catalyst mixture varied in the range of 1-7%. Zeolites have been used to reduce the amount of sulfuric acid used and increase selectivity for bio-polyol formation. The reusability of the zeolite catalyst was also assessed.

Finally, the resulting solvolysis products were characterised by determining the conversion of biomass, hydroxyl and acid count, respectively.

The biopolyols thus obtained were used in obtaining polyurethane products: foams, adhesives or coatings. The products were characterized by specific tests and comparisons were made with common commercial products obtained from petrochemicals.